**WO 2005/090836** 

10/5/5U/7 MONTO 15 SEP 2006 PCT/EP2005/002487

1

Sealing system for high-pressure and high-temperature containers

The present invention relates in general to the production of a sealing system for high-pressure and high-temperature containers and provides an apparatus and a method using this sealing system.

The apparatus to which the invention relates is more particularly of the type comprising:

- a container for high pressures defining a volume adapted to contain a material to be pressurised, provided with an inlet for the introduction of the material into this volume,
- a moving head adapted hermetically to close the pressurised container, and
- a piston mounted to slide in a leak-tight manner through the head and moving between a retracted position and a forward position in which it places the inner volume of the container under pressure.

This apparatus can be used to carry out technological processes which require particular high-pressure treatments, typically at more than 1000 bar, and at high temperatures, for instance of the order of 450-550°C.

The hot isostatic pressing (HIP) process is a particular instance of these technological processes; in this process, the container contains a bath of melted salts with a low melting point, in the liquid state, which is then pressurised by means of the piston associated with the head and which acts as a means for applying pressure to a casting immersed in a bath.

In known systems, the head may move between a raised open position and a lowered position, in which the head acts as a cover directly closing the inlet of the container, through the interposition of an annular sealing member between the respective facing surfaces; in the closed position, the seal between the piston and the head adapted to ensure appropriate sliding of the piston within the head is provided by means of at least one sealing member within the head.

For this purpose, bearing in mind the high temperature which - by conduction - occurs within the head and the high pressure that it is desired to generate, it is necessary to use gaskets formed from high-performance and high-cost materials, such as graphite gaskets.

When this sealing gasket has to be replaced after its deterioration, the head has to be dismantled and the plant shut down with a substantial increase in overall process management costs.

In order to remedy the above-mentioned drawbacks, the present invention relates to a sealing apparatus of the type described above, characterised in that it comprises an auxiliary cover, which can be associated in a leak-tight manner with the inlet of the high-pressure container, in a position interposed between the container and the head, this cover being provided with an opening in which the piston can slide and coinable sealing means associated with the opening in order to provide a pressurised seal between the piston and the cover in the position in which the moving head hermetically closes the container.

The moving head in particular comprises coining means adapted to cause the coining by plastic deformation of these sealing means, in its hermetic closure position.

The invention further relates to a method using the sealing apparatus described above, as set out in the following claims.

Further characteristic features and advantages of the apparatus and the method are set out in the accompanying claims.

The characteristic features and advantages of the apparatus and the method of the invention are set out in the following detailed description, made with reference to the accompanying drawings provided purely by way of non-limiting example, in which:

- Fig. 1 is a partially exploded section through an apparatus of the invention;
- Fig. 2 is a view in section through the apparatus of Fig.
- 1, in the closed configuration; and
- Fig. 3 is a view in section through a detail of the apparatus in relation to the coinable sealing means, the latter being shown before and after coining.

In the attached drawings, a high-pressure and high-temperature container of a type known per se is shown by 2 and internally contains a volume 4 adapted to contain a material to be pressurised, for instance a mass of salts of low melting point in the liquid state.

The container 2 has an upper inlet 6 whose contour is associated with a toroidal sealing gasket 8, for instance a copper gasket.

A head moving in the vertical direction  $F_1$  is shown by 10, a piston 12 being mounted to slide in this head.

A bushing 14, inserted in a seat 16 of the head and fixed therein by means of a closure plate 18, is used to ensure appropriate sliding of the piston within the head 10.

An annular flange insert 20 provided with an axial opening in which the piston can slide is associated with a seat 36 at the lower end of the head 10, facing the container 2.

The flange insert 20 is preferably formed by a material having better properties of mechanical and thermal strength than the material forming the head.

An auxiliary annular cover 22 moving vertically and/or horizontally is shown by 22 and is able to engage the contour of the inlet 6 of the container 2 in a leak-tight manner with the assistance of the gasket 8.

The auxiliary cover 22 has an axial opening 24, in which the piston 12 slides in a leak-tight manner.

The opening 24 has a seat 26, formed for instance by a widened section of the opening 24, in which an annular sealing gasket, of coinable material, i.e. capable of plastic deformation as a result of the pressure exerted by coining means 30 associated with the head 10, can be inserted.

WO 2005/090836 PCT/EP2005/002487

5

The coining means may in particular be formed by an annular formation 30 of the flange insert 20, which formation — in the position in which the annular insert is inserted into the seat 22 of the head 10 — projects downwards beyond the lower end surface 32 of the head and can be inserted into the coinable gasket seat 26. The coinable gasket may be formed by a copper or aluminium rod.

When the method to pressurise the container is actuated, the moving cover 22 is positioned to bear on the contour of the inlet 6 of the container 2, with a sealing gasket 8 which exerts a seal between the surface of the contour of the inlet of the container and the lower surface 34 of the cover.

The coinable gasket 28 is inserted in the seat 26. This closes the head 10 on the cover 22, bringing the head into the closed position shown in Fig. 2.

The lowering of the head into the closed position causes the coining of the gasket 28 which is plastically deformed and assumes the deformed configuration shown by 28a in Fig. 3.

In Fig. 3, the gasket 28 and the insert 20, with the relative coining means 30, are shown in dashed lines in the position immediately preceding the beginning of coining. The position of the insert 20, after coining, and the gasket in its deformed configuration, shown by 28a, in which this gasket has assumed the shape of the seat 26, are shown in continuous lines.

In the closed position, the gasket exerts an appropriate seal both in the axial direction of the piston and in the plane at right angles to the axis of the piston. The sliding of the piston within the head is ensured both by the bushing 14 and by appropriate plays between the piston 12, the flange insert 20 and the cover 22.

At this point, the method involves the pressurisation of the volume 4 within the container 2 by means of the descent of the piston and the maintenance of the pressure for the required cycle time.

Once the high-pressure heat treatment has taken place, the subsequent stages include the operation to raise and retract the piston and the movement of the head into the open position.

The movement of the auxiliary cover 22 may therefore take place by vertical and/or lateral displacement in order to enable the insertion of a new coinable seal and a further treatment cycle.

The method and the apparatus described above make it possible to obtain substantial advantages in their industrial application.

In particular, the coined gasket makes it possible to achieve adequate pressure and temperature sealing even at pressures of more than 1000 bar and high temperatures of the order of 450-550°C or more. The gasket can be particularly rapidly replaced with no need to shut down the pressurisation process. The replacement of the gasket may in particular be masked in the cycle time.

For this purpose, it will be appreciated that it is possible to use a second auxiliary cover 22, by removing the coined

WO 2005/090836 PCT/EP2005/00248

7

gasket used in a preceding cycle and positioning a new coinable gasket during the pressing cycle time, in which a first auxiliary cover is used; this characteristic feature is particularly advantageous in hot isostatic pressing processes, in which the treatment cycle time is typically in the range of 15-40 seconds.

Moreover, coinable gasket materials which are of low cost and suitable for recycling, for instance copper or aluminium materials, can be used.

It will be appreciated that the invention is not limited to the use of these gasket materials, but also includes the use of other materials, provided that they enable conditions in which the piston may slide when coining has taken place.